2.4 Particle-Size Analysis

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2.4.1 Introduction

Particle-size analysis (PSA) is a measurement of the size distribution of individual particles in a soil sample. The major features of PSA are the destruction or dispersion of soil aggregates into discrete units by chemical, mechanical, or ultrasonic means and the separation of particles according to size limits by such means as sieving and sedimentation, as well as other methods (diffraction).

Soil particles span a large size range, varying from stones and rocks (exceeding 0.25 m in size) down to submicron clays (<1 µm). Various systems of size classification have been used to define arbitrary limits and ranges of soil-particle size. Soil particles smaller than 2000 µm are generally divided into three major size groups: sands, silts, and clays. These groups are sometimes called *soil separates* and can be subdivided into smaller size classes. Figure 2.4–1 shows the particle size, sieve dimension, and defined size class for the system of classification used by the U.S. Department of Agriculture (USDA), the Canadian Soil Survey Committee (CSSC), the International Soil Science Society (ISSS), and the American Society for Testing and Materials (ASTM). Geologists and geomorphologists typically use the Wentworth classification scheme (Wentworth, 1922) and that of Folk (1954), and variations of the Folk scheme (Folk, 1980; Prothero & Schwab, 1996). The Soil Science Society of America has adopted the USDA classification, that is: sands (<2000–50 µm), silts (<50–2 µm), and clays (<2 µm). Although the USDA classification scheme will be emphasized in the following methods, it should be recognized that other systems are frequently cited, particularly in engineering literature; hence, when reporting results, care should be taken to specify clearly which system is being used.

Particle-size analysis data can be presented and used in several ways, the most common being a cumulative particle-size distribution curve. An example is shown in Fig. 2.4–2. The percentage of particles less than a given particle size is plotted against the logarithm of the effective particle diameter. Particle-size distribution curves, when differentiated graphically, produce frequency distribution curves for various particle sizes. Frequency curves usually exhibit a peak or peaks representing the most prevalent particle sizes.

Particle-size distribution curves are used extensively by geologists in geomorphological studies to evaluate sedimentation and alluvial processes, and by civil engineers to evaluate materials used for foundations, road fills, and other con-

struction purposes. Details of the use of these curves are given by Krumbein and Pettijohn (1938) and Irani and Callas (1963). Skaggs et al. (2001) have developed a useful method to estimate particle-size distribution curves from limited textural data.
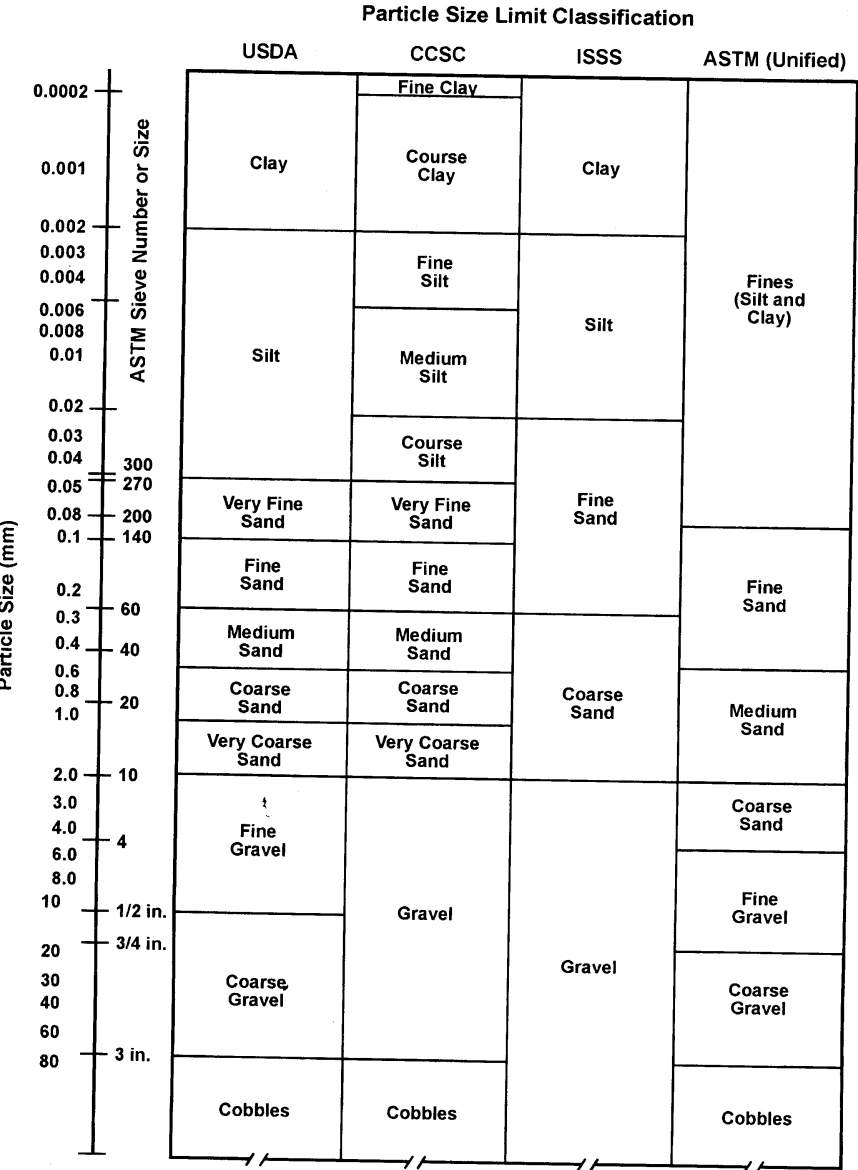
Particle Size Limit Classification

Particle Size (mm)	ASTM Sieve Number or Size	USDA	CCSC	ISSS	ASTM (Unified)
0.0002			Fine Clay		
0.001		Clay	Course Clay	Clay	
0.002					
0.003 / 0.004		Silt	Fine Silt		Fines (Silt and Clay)
0.006 / 0.008 / 0.01				Silt	
0.02			Medium Silt		
0.03 / 0.04	300		Course Silt		
0.05	270				
0.08	200	Very Fine Sand	Very Fine Sand	Fine Sand	
0.1	140				
0.2		Fine Sand	Fine Sand		Fine Sand
0.3	60				
0.4	40	Medium Sand	Medium Sand		
0.6 / 0.8	20	Coarse Sand	Coarse Sand	Coarse Sand	Medium Sand
1.0					
		Very Coarse Sand	Very Coarse Sand		
2.0	10				
3.0		Fine Gravel	Gravel	Coarse Sand	Coarse Sand
4.0 / 6.0	4				
8.0					Fine Gravel
10	1/2 in.				
20	3/4 in.	Coarse Gravel		Gravel	
30 / 40					Coarse Gravel
60					
80	3 in.				
		Cobbles	Cobbles		Cobbles

Fig. 2.4–1. Particle-size limits according to several current classification schemes. USDA, U.S. Department of Agriculture (Soil Survey Staff, 1975); CSSC, Canada Soil Survey Committee (McKeague, 1978); ISSS, International Soil Sci. Soc. (Yong & Warkentin, 1966); ASTM (Unified), American Society for Testing and Materials (ASTM, D-2487, 2000a).

Particle-size analysis is often used in soil science to evaluate soil texture. Soils rarely consist entirely of one size range. Soil texture is based on different combinations of sand, silt, and clay separates that make up the particle-size distribution of a soil sample. Figure 2.4–3 shows the USDA-defined limits for the basic soil textural classes. Details for interpreting the textural triangle for soil-classification purposes are given by the Soil Survey Staff (1975). The ASTM (Unified) engineering classification system is used widely for delineating soil types for construction purposes (Fig. 2.4–4). In this system, liquid limits and plasticity indexes must be known to properly classify the soil type (American Society for Testing and Materials [ASTM], 2000a, b).

Hydrologists often use PSA as a means of predicting hydraulic properties, particularly for sands (Todd, 1964; Bloemen, 1980). Arya and Paris (1981) and Arya et al. (1999a, b) have used PSA combined with bulk density information as a means to predict water retention and unsaturated hydraulic conductivity of soils for materials ranging from sands to clays. These predictive methods appear to work best on minimally aggregated soil materials. Where aggregation is important to the hydraulic properties, both particle-size and aggregate-size distributions are required for the analysis (see Section 2.6 and Nimmo, 1997).

2.4.2 Pretreatment and Dispersion Techniques

2.4.2.1 General Principles

Pretreatment of samples to enhance the separation or dispersion of aggregates is a key step in PSA and is generally recommended since many soils contain aggregates that are not readily dispersed. Soils generally contain organic matter and often contain Fe oxides and carbonate coatings that bind particles together. Chem-
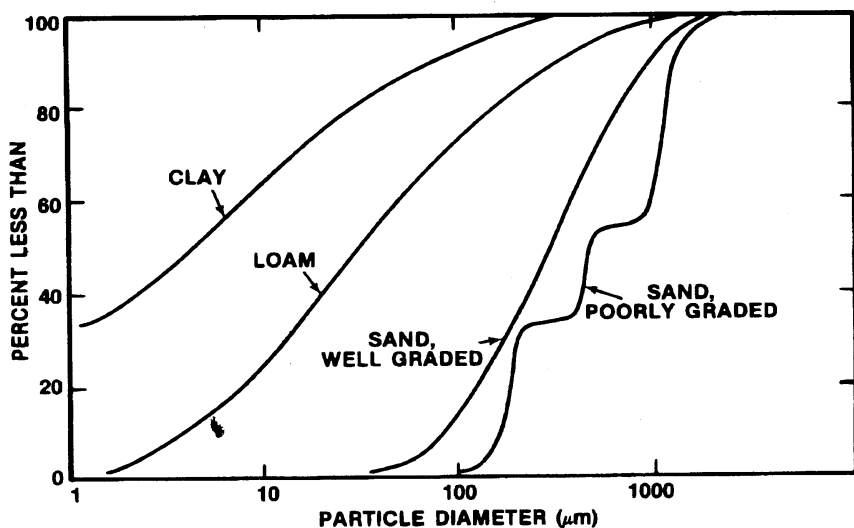

Fig. 2.4–2. Particle-size distribution curves for several soil materials (after Hillel, 1982).
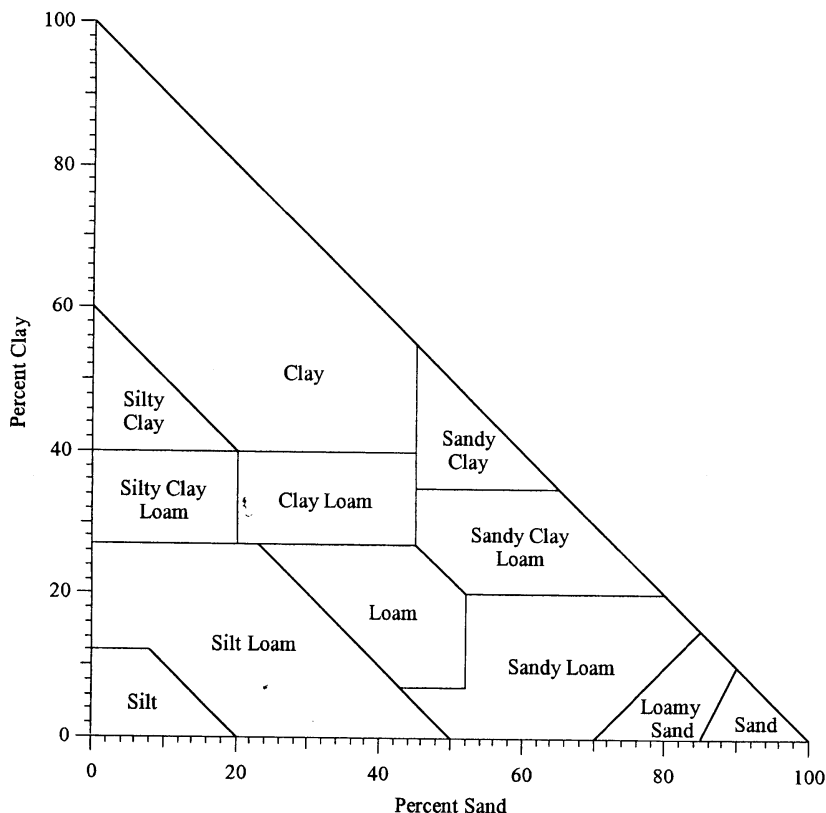
Fig. 2.4–3. Textural triangle for soil textural analysis using the USDA classification scheme.

ical pretreatments are used to remove these coatings; however, chemical treatment can destroy and dissolve some soil minerals. Physical treatments are also used, but standardization of treatment and adequate testing of specific methods are needed since the very process of separation by mechanical or ultrasonic means can fragment the individual particles into further subunits. Procedures should clearly specify the sample pretreatment, the separation method, and the purpose for which the size analysis is intended for a particular soil.

Standard PSA methods require that soil particles be dispersed in an aqueous solution by both chemical and physical means. After pretreatment, chemical dispersion is often accomplished using a dilute alkaline solution of sodium polyphosphate. The effectiveness of the chemical dispersing agent depends on its ability to create and maintain repulsive forces between soil particles. Some soils (e.g., those of volcanic ash origin) that have been highly weathered disperse more readily in acid media; hence, some pretesting may be required to determine the effects of soil mineralogy and other factors on soil dispersibility and to select an appropriate method to achieve complete dispersion. Physical dispersion of particles is accomplished by shearing action or turbulent mixing, using mechanical shakers, electrical mixers, or ultrasonic probes.

Dispersibility of soils low in organic matter depends primarily on soil mineralogy. Highly oxidized soils are particularly difficult to disperse. Examples include the "subplastic" soils of Australia (McIntyre, 1976; Brewer & Blackmore, 1976; Walker & Hutka, 1976; Blackmore, 1976; Norrish & Tiller, 1976). Depending on the method of chemical treatment and physical dispersion used, measured clay content for an individual soil sample can vary by factors of four or more.

Volcanic-ash soils are high in amorphous (noncrystalline) clay-sized materials and have great resistance to dispersion, particularly after air or oven drying (Kubota, 1972; Schalscha et al., 1965; Espinoza et al., 1975; Maeda et al., 1977). Kubota (1972) reported clay contents ranging from 1 to 56% (w/w) for one volcanic-ash soil, depending on pretreatment. Maximum clay content was obtained when the soil was retained at field moisture before ultrasonic dispersion. Warkentin and Maeda (1980) recommend that volcanic-ash soils be left at field moisture and dispersed at either pH 3 or above pH 9. Tama and El-Swaify (1978) and El-Swaify (1980) have observed that soils with variable charge are particularly difficult to disperse unless the dispersant solution is well below or above the zero point of charge.

Highly aggregated, stable clay soils may behave like coarse sands in terms of water infiltration; hence, they may be identified in the field as sands or coarse loams. These same soils, having significant microporosity and high exchange capacities, retain water and nutrients much better than sands. For agricultural purposes, these soils should be texturally classed in a much finer category than they appear in the field. For soils where these uncertainties are known to exist, measurements such as a simple dispersive index (Sherard et al., 1976), ASTM dispersion test (ASTM, 2000c), or the water-stability of aggregates (see Section 2.6) would be necessary and useful information. Also, a calculated clay content, determined from a ratio of the cation-exchange capacity (CEC), CEC of the total soil/CEC of the clay-size material (Norrish & Tiller, 1976), can be used to estimate the theoretical maximum clay fraction of the soil material.

The method that produces the most complete dispersion of a soil sample is generally the more acceptable method. However, the chemical treatment and mechanical work done on the soil are dictated by somewhat arbitrary decisions, so there is no absolute size distribution for a given sample. Intense mechanical or ultrasonic dispersion, coupled with appropriate chemical treatment, should yield a sample with most of the clay minerals in the measured clay fraction. In contrast, a less drastic chemical treatment and/or little mechanical dispersion may reflect the more natural particle-size distribution of the soil. Comparisons of PSA results should always include comparisons of the pretreatment and dispersion methods used.

2.4.2.2 Organic Matter Removal

2.4.2.2.a Principles

Removing organic matter is often a first step in the chemical pretreatment of many soils. The necessity and difficulty of removing organic matter depends on the intended use of the analytical results of the PSA, the nature and concentration of organic matter in the sample to be analyzed, the pH of the soil, and the associated presence in the soil of free carbonates, gypsum, oxides, and soluble salts.

			Criteria for assigning group symbols and group names using laboratory tests[A]		Soil classification	
					Group symbol	Group name[B]
Coarse-grained soils (More than 50% retained on No. 200 sieve)	Gravels (>50% of coarse fraction retained on No. 4 sieve)	Clean gravels (<5% fines[C])	$Cu > 4$ & $1 < Cc < 3$[E]		GW	Well-graded gravel[F]
			$Cu < 4$ and/or $1 > Cc > 3$[E]		GP	Poorly graded gravel[F]
		Gravels with fines (>12% fines[C])	Fines classify as ML or MH		GM	Silty gravel[F,G,H]
			Fines classify as CL or CH		GC	Clayey gravel[F,G,H]
	Sands (50% or more of coarse fraction passes No. 4 sieve)	Clean sands (<5%)	$Cu > 6$ & $1 < Cc < 3$[E]		SW	Well-graded sand[I]
			$Cu < 6$ and/or $1 > Cc > 3$[E]		SP	Poorly graded sand[I]
		Sands with fines (>12% fines[D])	Fines classify as ML or MH		SM	Silty sand[G,H,I]
			Fines classify as CL or CH		SC	Clayey sand[G,H,I]
Fine-grained soils (50% or more passes the No. 200 sieve)	Silts and clays (Liquid limit <50)	Inorganic	$PI > 7$ & plots on or above "A" line[J]		CL	Lean clay[K,L,M]
			$PI < 4$ or plots below "A" line[J]		ML	Silt[K,L,M]
		Organic	Liquid limit—oven-dried / Liquid limit—not dried	<0.75	OL	Organic clay[K,L,M,N] / Organic silt[K,L,M,O]
	Silts and clays (Liquid limit 50 or more)	Inorganic	PI plots on or above "A" line		CH	Fat clay[K,L,M]
			PI plots below "A" line		MH	Elastic silt[K,L,M]
		Organic	Liquid limit—oven-dried / Liquid limit—not dried	<0.75	OH	Organic clay[K,L,M,P] / Organic silt[K,L,M,O]
Highly organic soils	Primarily organic matter, dark in color, and organic odor				PT	Peat

[A]Based on the material passing the 75-mm (3-in.) sieve. [B]If field sample contained cobbles or boulders, or both, add "with cobbles or boulders, or both" to group name. [C]Gravels with 5–12% fines require dual symbols: GW-GM, well-graded gravel with silt; GW-GC, well-graded gravel with clay; GP-GM, poorly graded gravel with silt; GP-GC, poorly graded gravel with clay. [D]Sands with 5–12% fines require dual symbols: SW-SM, well-graded sand with silt; SW-SC, well-graded sand with clay; SP-SM, poorly graded sand with silt; SP-SC, poorly graded sand with clay. [E]$Cu=D_{60}/D_{10}$ and $Cc= (D_{30})^2 D_{10} \cdot D_{60}$. [F]If soil contains ≥15% sand, add "with sand" to group name. [G]If fines classify as CL-ML, use dual symbol GC-GM, or SC-SM. [H]If fines are organic, add "with organic fines" to group name. [I]If soil contains ASTM 15% gravel, add "with gravel" to group name. [J]If Atterberg limits plot in hatched area, soil is a CL-ML, silty clay. [K]If soil contains 15–29% plus No. 200, add "with sand" or "with gravel," whichever is predominant. [L]If soil contains ASTM 30% plus No. 200, predominantly sand, add "sandy" to group name. [M]If soil contains ASTM 30% plus No. 200, predominantly gravel, add "gravelly" to group name. [N]PI ASTM 4 and plots on or above "A" line. [O]PI < 4 or plots below "A" line. [P]PI plots on or above "A" line. [Q]PI plots below "A" line.

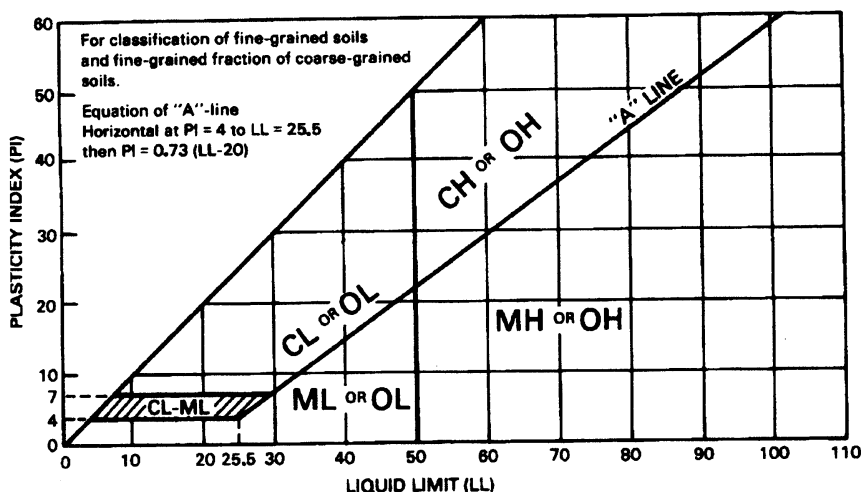Fig. 2.4–4. Unified soil classification system, including plasticity chart (ASTM, 2000a).

For classification of fine-grained soils and fine-grained fraction of coarse-grained soils.

Equation of "A"-line
Horizontal at PI = 4 to LL = 25.5
then PI = 0.73 (LL-20)

(Chart axes: PLASTICITY INDEX (PI) from 0 to 60; LIQUID LIMIT (LL) from 0 to 110. Regions labeled: CH OR OH, "A" LINE, MH OR OH, CL OR OL, ML OR OL, CL-ML)

Fig. 2.4–4. Continued.

2.4.2.2.b Method

A variety of reagents have been used in the past to successfully remove organic matter. Notable among these are hydrogen peroxide (H_2O_2), sodium hypochlorite, sodium hypobromite, and potassium permanganate. Hydrogen peroxide has been recommended as the standard oxidant for most soils (Day, 1965).

2.4.2.3 Iron Oxide Removal

2.4.2.3.a Principles

Coatings and crystals of various Fe oxides, such as hematite and goethite, often act as cementing and binding agents in soils. Removing these cementing agents aids in dispersing the silicate portion of the soil and is often necessary for accurate mineralogical analysis.

2.4.2.3.b Method

Mehra and Jackson (1960) recommend the use of a bicarbonate-buffered, sodium dithionite-citrate system to remove Fe oxide. This method, compared with several other methods for removing free Fe oxides from latosolic soils, was found to be the most effective. In addition, this method was the least destructive of Fe silicate clays, as indicated by the least loss of cation-exchange capacity. Mehra and Jackson (1960) indicated that the optimum pH for maximum Fe oxide removal was approximately 7.3. Since considerable OH^- is expended in the sodium dithionite-citrate reaction with Fe oxide, a buffer is needed to hold the pH at the optimum level. Sodium bicarbonate has proven to be an effective buffer. This procedure minimizes the formation of sulfide, Fe sulfide, Zn oxalate, or other unwanted precipitates during Fe oxide removal.

In soils where Fe oxides are part of the dominant mineralogy, it is not recommended that Fe oxides be removed since many of the primary mineral grains in the clay fraction could be destroyed (El-Swaify, 1980).

2.4.2.4 Carbonate Removal

2.4.2.4.a Principles

Carbonates, principally Ca and Mg, can cause aggregation in soils, and unless they are removed, it is difficult to achieve any meaningful separation of silt and clay due to the continual breakdown of the carbonate particles (Kunze & Dixon 1986). Carbonates may be removed by heating and peroxide treatment, with sodium acetate buffer, or with disodium dihydrogen ethylenediaminetetraacetate. Soluble salts may be removed as part of the same treatment.

2.4.2.4.b Method

Carbonate can be removed relatively easily from soils before dispersion and sedimentation by acidifying the sample. Heating accelerates the reaction. Samples that are acidified before organic matter removal with H_2O_2 will usually be free of carbonates. Hydrogen chloride (HCl) treatment can destroy the crystalline lattice of clay minerals; therefore, acid treatment with 1 M NaOAc at pH 5 is preferred.

2.4.2.5 Soluble Salts Removal

2.4.2.5.a Principles

A variety of soluble salts, including sodium, calcium, and magnesium chlorides and carbonates, are commonly found in alkaline soils. High concentrations of soluble salts can cause flocculation of soil suspensions. Alkaline salts can cause decomposition of H_2O_2, decreasing its effectiveness as an oxidizing agent for soil organic matter. In addition, many soluble salts interfere with saturation of the exchange complex. Calcium and magnesium salts, commonly occurring as carbonates, are relatively unstable and are often measured as part of the clay and silt fractions.

2.4.2.5.b Method

The most common procedure for removing soluble salts is to leach the salts with distilled water. Samples can be washed with distilled water with a filter candle or by centrifuging. The procedure should be repeated until the leachate salt concentration drops below 10 mM. The washing treatment is then followed by chemical and physical dispersion.

2.4.2.6 Sample Dispersion

Soils can be dispersed with a combination of methods. The methods for dispersion can be classified as either chemical or physical. Numerous methods of chemical dispersion have been investigated and reported (Theisen et al., 1968; Norrish & Tiller, 1976). Soils are chemically dispersed after oxidizing organic matter and removing carbonates and Fe oxides. Chemical dispersion is based primarily on the concept of particle repulsion as a result of elevation of the particle electrokinetic (zeta) potential (Soil Science Society of America, 1996). This process is usually

accomplished by saturating the exchange complex with Na. Physical or mechanical methods of dispersion involve separating the individual particles by means of some mechanical or physical process, such as rubbing, rolling, shaking, or vibrating. During the past 20 yr, electronic dispersion, primarily by the use of ultrasonics, has become increasingly popular. Most researchers have found that a combination of chemical and physical or electronic methods provides the most complete and stable dispersion (Maeda et al., 1977; Mikhail & Briner, 1978).

2.4.2.6.a Chemical Dispersion

Principles. Following removal of cementing and flocculating agents, samples must be dispersed and maintained in a dispersed state until sedimentation measurements are completed.

Method. A number of dispersing chemicals have been used. These include Na-hexametaphosphate (HMP), Na_2PO_7, NaOH, Na_2CO_3, and NaOBr. Of these, HMP appears to be the most commonly used dispersant. Commercial detergents contain quantities of HMP and other soluble phosphates, but uncertainty exists as to the exact amounts (Yaalon, 1976; Veneman, 1977). For this reason, reagent-grade HMP, which is commercially available, is the recommended chemical dispersant for the pipette and hydrometer tests described later in this chapter.

The exact amount of chemical dispersant needed to prevent flocculation depends on soil type (mineralogy and other characteristics). Flocculation often can be prevented by increasing the concentration of the dispersant solution. It should be noted that the pipette analysis requires only 0.5 g L^{-1} HMP, compared with a 5 g L^{-1} HMP solution for the hydrometer analysis. The lower amount needed for pipette analysis is probably due to pretreatment (organic matter, Fe oxide, and soluble salt removal). Specific amounts used in these analyses have been established by empirical methods.

2.4.2.6.b Physical Dispersion

Principles. Several methods of physical dispersion have been used in conjunction with pretreatment and chemical dispersion.

Method. The ASTM (2000d) recommends either an electric mixer with specially designed stirring paddles or an air-jet stirrer (Chu & Davidson, 1953; Theisen et al., 1968). For the hydrometer method, Day (1965) recommends a 5-min mixing with a standard electrical mixer (malted milk style), but cautions that the mixer blades deteriorate rapidly by abrasion and should be replaced after 1 or 2 h of use or when showing signs of wear. Reciprocating shakers have also been used. Overnight shaking is prescribed in the pipette procedure and can be used in the hydrometer method. However, the larger sample (40 g) used in the hydrometer method will pack to the bottom of 250-mL bottles; hence, larger (>500-mL) shaking bottles are recommended for the larger samples to avoid this problem. High-speed reciprocating shakers have been used effectively on small samples of 10 g or less (El-Swaify, 1980). These high-speed shakers optimize dispersion when the liquid/solid ratio is about 5:1.

2.4.2.6.c Ultrasonic Dispersion

Principles. The principle behind ultrasonic dispersion is the transmission of vibrating sound waves in the soil solution. The sound waves produce microscopic bubbles, which collapse, producing cavitation. The release of intense energy of cavitation literally blasts the soil aggregates apart, causing dispersion even in highly aggregated soils.

Method. Much work has been done in testing the use of ultrasonic dispersion of soils, but no standard procedures have been adopted (Edwards & Bremner, 1964, 1967; Saly, 1967; Bourget, 1968; Watson, 1971; Kubota, 1972; Mikhail & Briner, 1978). An initial concern with this method of dispersion was the possible destruction of primary particles, but Saly (1967) reported that ultrasonic vibration did not destroy the crystalline lattice or break down the primary grains. Edwards and Bremner (1964, 1967) investigated the use of ultrasonic dispersion in the absence of a dispersing agent. For mineralogical analysis, ultrasonic dispersion was preferred since dispersion was achieved without pretreating the soil or adding a dispersing agent.

Edwards and Bremner summarized the following advantages of ultrasonic dispersion:

1. The resultant suspension is stable; hence, flocculation does not occur during sedimentation.
2. The method works well for dispersing calcareous soils, organic soils, and soils with high clay content.
3. Ultrasonic dispersion does not destroy organic matter.
4. Ultrasonic dispersion does not alter the soil pH, electrical conductivity, or cation exchange capacity.

In contrast to the work of Edwards and Bremner, Mikhail and Briner (1978) reported that the most satisfactory method of pretreatment and dispersion involved the following steps: oxidation of organic matter, removal of carbonates and acid washing, and Na saturation followed by ultrasonic dispersion. The results indicated that the highest degree of dispersion was achieved by this technique. Kubota (1972) reported that a sonic dispersion at low pH was effective in dispersing peroxide-treated volcanic-ash soils. Each of the above authors used a different ultrasonic power and dispersion time, indicating that effective dispersion with ultrasonics is soil dependent. For routine PSA, no standard method for ultrasonic mixing is proposed at this time. Much additional research is needed to determine the effectiveness or limitations of ultrasonic dispersion for a wide range of soil materials.

2.4.3 Specific Methods of Particle-Size Analysis

2.4.3.1 Introduction

Three methods are described in detail in the following sections: sieving, pipette, and hydrometer. In addition, a brief discussion of laser-diffraction techniques and pressure measurement is also provided. The pipette and hydrometer methods

fall under the category of gravitational sedimentation methods, and there is a general introduction to these methods.

2.4.3.2 Analysis by Sieving

2.4.3.2.a Principles

The typical particle-size range for sieving is 50 to 2000 μm. Several limitations of sieving have been noted in the past. Day (1965) indicated that the probability of a particle passing through a sieve in a given time of shaking depends on the nature of the particle, the number of particles of that size, and the properties of the sieve. Particle shape and sieve-opening shape affect the probability of passage. For example, a particle whose shape permits its passage only in one orientation has a limited chance of getting through, except after prolonged shaking. Sieve openings are generally unequal in size, and extensive shaking is required before all particles have had the opportunity of approaching the largest openings. In fact, it is rare that complete sorting of a given size range can be achieved. Good reproducibility requires careful standardization of procedure.

Load and Particle Shape Effect on Sieving Time. The sieving process involves two primary regions: (i) an initial region involving the passage of particles much finer than mesh openings and (ii) a region related to passage of near-mesh particle sizes (Allen, 1997). Aslan et al. (1998) refined the proportional model of Kaye (1962) for relating the probability of particle passage at time t to the particle mass on the sieve that would eventually pass through:

$$dM_t/dt = -k(M_t - M_r) \qquad [2.4-1]$$

where M_t is the total mass of material on the sieve at time t, M_r is the material mass that does not pass the sieve, and k is the passage-rate factor. The modification developed by Aslan et al. (1998) was based on evidence showing that the rate coefficient k is also time dependent. They proposed a parametric form: $k = ct^{-m}$ that, upon integration of Eq. [2.4–1], yields:

$$M = M_0[1 - \exp(-ct^n)] \qquad [2.4-2]$$

where M_0 is the particle mass that would pass through the sieve at infinite sieving time; M is the cumulative mass of material passed at time t; and $n = 1 - m$. Denoting the initial particle mass on the sieve as M_T, we can express M as $M = M_T - M_t$, with $M_T = M_0 + M_r$. It is interesting to note that Eq. [2.4–4] is known as the *Weibull distribution*, commonly applied to characterization of "time to failure" in several engineering problems (Aslan et al., 1998). The Weibull distribution is also used to characterize fracturing processes similar to those responsible for the generation of particle-size distribution (Technov & Yanev, 1986; Assouline et al., 1998).

The results of Aslan et al. (1998) demonstrate that increasing the fraction of oversized particles (diluent) on the sieve reduces sieving efficiency and thus increases the time for passage. Experimentally, it was shown that increasing the diluent fraction reduces the values of the parameters c and n. The particle shape also

Table 2.4–1. Effects of load and sieving time on amount of material passing 80-μm (200-mesh) sieve (Allen, 1997).

	Sieving time (min)			
	5	10	20	40
Sample weight	Retained on sieve			
g	%			
500	83.6	80.7	76.5	73.8
250	67.2	64.3	61.6	57.8
125	58.6	58.0	55.2	53.2
62.5	56.6	55.0	53.2	52.3

affected the value of n, with $n = 2$ for smooth glass beads and $n = 1.2$ for rough sand particles (Aslan et al., 1998). Table 2.4–1 summarizes experimental results of Shergold (1946) as presented in Allen (1997) for the effect of sieving time and load on the amount of sand passing the 80-μm (200-mesh) sieve (Fig. 2.4–5).
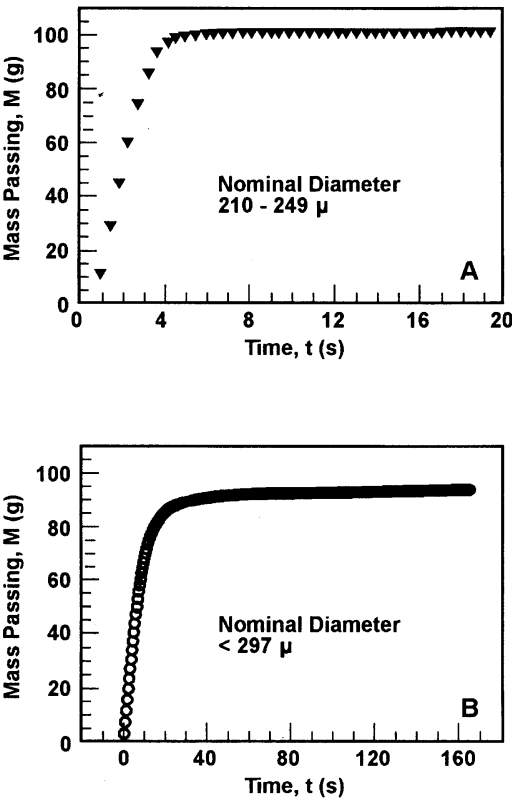

Fig. 2.4–5. Sieving curves for (A) glass beads and (B) sand sieved in a 297-μm (50-mesh) square wire sieve. Both samples have a nominal mass of 110 g (Aslan et al., 1998).

Considering sieve apertures as gauges that reject or pass particles as they are presented to the aperture suggests that the probability of a particle presenting itself to the aperture is dependent on (Allen, 1997):

1. The particle-size distribution of the sample—a large fraction of near-mesh size reduces efficiency.
2. The load on a sieve (or number of particles)—a smaller load passes faster.
3. The physical properties of the material—affects the likelihood for aggregation, adhesion to the sieve.
4. The method of sieve shaking or other motion affects the potential for aperture blockage and should involve some jolting of particles wedged in the sieve mesh.
5. Particle shape—elongated particles pass more slowly than round particles.
6. Sieving surface geometry—including fraction of open area, angle.

These factors highlight the dependence on particle shape and the capability to attain a proper orientation within the time allotted for sieving. These effects often result in an apparent coarsening of the particle population relative to their "true" sieve size (Syvitski, 1991). In general, the sieve size is a combined measure of the least cross-sectional area and the maximal axial length. For particles with equal cross-sectional area, those with longer axial length are more likely to be trapped on a sieve.

The ϕ Notation. A scaling scheme, expressed in terms of the parameter, ϕ, often used in the sedimentology literature for representation of particle-size distribution, is given by the following transformation:

$$X_i = X/X_o \qquad\qquad [2.4\text{--}3]$$

where X_i is a dimensionless ratio of a given particle size, X (mm), to standard particle size, X_o, of 1 mm. The relationships, $\log(X) = \phi/\log 2 = 3.322\phi$, are used to find the particle size; for example, $\phi = 8.966$ represents a value of $X = 0.002$ mm ($= 2$ μm). The standard deviation used in this scaling scheme is defined as:

$$\sigma_\phi = (1/2)(\phi_{84} - \phi_{16}) \qquad\qquad [2.4\text{--}4]$$

where ϕ_{84} and ϕ_{16} are the particle sizes for the 16th and 84th percentiles.

2.4.3.2.b Apparatus for Sample Preparation

1. Drying trays
2. Wooden rolling pin
3. Na–HMP solution (50 g L^{-1})
4. Sieves. Large 20.5-cm (8 in.) diameter sieve with a 2-mm (2000-μm) square-hole screen. Other screen sizes needed include 5, 20, and 75 mm (USDA, 1982); 5 mm (#4),13 mm (1/2 in.), 20 mm (3/4 in.), 25 mm (1 in.), 50 mm (2 in.), and 75 mm (3 in.) (ASTM, 2000d).
5. Ruler or caliper capable of measuring to 250 mm (10 in.)

2.4.3.2.c Sample Preparation Method

Spread the bulk sample thinly (in 2- to 3-cm-thick layers, maximum) on trays and air-dry. Thoroughly mix and roll the sample with a wooden rolling pin to break up clods to pass a 2-mm sieve. Sieve out the >2-mm size fractions. Continue rolling and sieving until only coarse fragments that do not slake in water or HMP solution remain on the 2-mm screen. Use a rubber roller for samples with easily crushed coarse fragments. Sieve larger size fractions, record weights, and use total sample weight to calculate the percentage of total sample <2 mm.

2.4.3.2.d Comments

Sometimes it is desirable to keep the sample at field moist conditions. If this is determined appropriate, force the field moist sample through the 2-mm screen by hand, using a large rubber stopper, double bag the sample in plastic, and store for further use. From a separate subsample, determine the water content so that a check can be made on possible drying effects during storage.

Whether material >2 mm in diameter is sieved depends on the purpose for the data set. For soil-survey purposes, methods specified by the USDA (1982) may be used. For engineering purposes, the material >2 mm can be sieved according to requirements specified by ASTM method D-2487 (ASTM, 2000a).

Sample size depends upon the maximum size fragments present. Suggested sample sizes are:

1. Particles up to 20 mm in diameter—use 5 kg or more.
2. Particles up to 75 mm in diameter—use 20 kg or more.
3. Particles up to 250-mm diameter—use 100 kg or more.

Because of the large samples required, the volume percentage of particles coarser than about 20 mm is usually estimated. A suggested procedure for handling coarser fragments follows.

Weigh and sieve the entire sample through 75- and 20-mm screens. Weigh the >75-mm and the 75- to 20-mm fractions. Take a subsample of the <20-mm fraction for laboratory processing. Weigh the <20-mm sample before and after air-drying and correct the total sample weight for the loss of water from field conditions. Separate and weigh the 2- to 5-mm and the 5- to 20-mm fractions. If fine earth adheres to the coarse fraction, wash the coarse material, dry, reweigh, and apply the appropriate corrections. Calculate the coarse fractions as a percentage of the <20-mm material (or the <75-mm, or the <250-mm, depending upon the size limit involved in sampling). Note that for taxonomic (classification) purpose, stones or rock fragments >250 mm (10 in.) are separated and used to estimate the volume of coarse fragments for family placement of soils. A large caliper or ruler can be used to check the dimensions of the >250-mm material. In addition, weight measurements and volume-displacement techniques can be used to evaluate coarse-fragment volume.

Sieving of materials <2000 μm (2 mm) depends on the size separations required, but should always be done as a wet-sieve procedure because dry materials <2000 μm tend to aggregate. Dry sieving of soils containing any silt or clay size particles will always underestimate the primary particle sizes. Specific sieving

procedures are described in the sections on pipette (Section 2.4.2.4.c) and hydrometer (2.4.3.5.c) methods.

2.4.3.3 Analysis by Gravitational Sedimentation

Sedimentation analysis relies on the relationship that exists between settling velocity and particle diameter. Settling velocity is related to the diameter of a spherical particle in the following way. The force acting downward on each particle due to its weight in water is

$$F_{\text{down}} = 4/3 \ \pi(X^3/8)(\rho_s - \rho_l)g \qquad [2.4\text{--}5]$$

where X is the particle diameter, ρ_s is the particle density, ρ_l is the liquid density, and g is the acceleration due to gravity.

Since this equation and those that follow (2.4–6 through 2.4–13) are not empirical, SI units can be used, but most often mixed units (e.g., g, cm, poise) are more conventional.

Because of the viscous resistance of the water, the opposing upward force is

$$F_{\text{up}} = 3\pi X \eta v \qquad [2.4\text{--}6]$$

where η is the fluid viscosity and v is the velocity of fall. The resisting force is zero where velocity, v, is zero at time $t = 0$, and it increases with increasing v until it is equal to the downward force. For sedimenting particles in a dilute dispersant solution, it can be shown that the terminal velocity for silt- and clay-size particles is reached in a relatively short time (a few seconds).

Equating F_{down} and F_{up} relates the terminal velocity to the particle diameter as follows:

$$v = [g(\rho_s - \rho_l)X^2]/18h \qquad [2.4\text{--}7]$$

A form of this relationship was first developed by Stokes (1851) and is now known as *Stokes' Law*. Basic assumptions used in applying Stokes' Law to sedimenting soil suspensions are:

1. Terminal velocity is attained as soon as settling begins.
2. Resistance to settling is entirely due to the viscosity of the fluid.
3. Particles are smooth and spherical.
4. There is no interaction between individual particles in the solution.

Gibbs et al. (1971) have shown that Assumptions 1 and 2 are met by soil particles <80 µm in diameter (sample calculations below provide a means to assess these for other particle sizes). Since soil particles are not smooth and spherical, X must be regarded as an equivalent rather than actual diameter. The assumptions of Stokes' Law as applied to soils are discussed fully by Krumbein and Pettijohn (1938).

In mineralogical analysis, there is often a need to separate various clay fractions for specific analysis. The clay fraction can be removed by sedimentation by

homogenizing a soil suspension and decanting all that remains above the plane $z = -h$ after time, t, where

$$t = 18\eta h/[g(\rho_s - \rho_l)X^2] \qquad [2.4-8]$$

Quantitative separation by decanting requires that the residue be resuspended and decanted repeatedly to salvage those particles that were not at the top of the suspension at the start of the sedimentation period.

Time and Distance for Attainment of Terminal Velocity. To evaluate certain experimental systems, it is instructive to estimate the time and distance required for a spherical particle to attain terminal velocity. The force balance equation before attainment of a terminal velocity (i.e., for $dv/dt \neq 0$) is:

$$(\pi/6)(\rho_s - \rho_l)gX^3 - 3\pi\eta Xv = (\pi/6)\rho_s X^3 (dv/dt) \qquad [2.4-9]$$

Note the inertial term on the left-hand side resulting from the force imbalance. Separation of variables and integration yields:

$$v(t) = \frac{(\rho_s - \rho_l)gX^2}{18\eta} \left[1 - \exp\left(-\frac{18\eta}{\rho_s X^2} t \right) \right] \qquad [2.4-10]$$

as $t \rightarrow \infty$, the velocity approaches the Stokes velocity (Eq. [2.4–3]). The time it takes a spherical soil particle with $X = 5$ μm to reach 99% of its terminal velocity in aqueous solution is about 0.017 ms, and for $X = 1000$ μm, the time is 0.68 s. The distance traveled by a particle for attainment of 99% terminal velocity,

$$h(t) = \frac{0.785(\rho_s - \rho_l)gX^2}{18\eta} t \qquad [2.4-11]$$

is also very small because the 99% attainment time is short. Hence, for all practical purposes, attainment of terminal velocity for sedimentation method in aqueous solutions is instantaneous.

Effects of Particle Shape. Soil particles are heterogeneous and asymmetrical. Unlike the steady settlement of symmetrical particles, asymmetrical particles tend to drift and wobble without attaining a fixed orientation or a constant velocity. Nonspherical particles invariably have higher resistance than a sphere, and thus a reduced settling velocity relative to a sphere of equal volume or surface area (Komar & Reimes, 1978; Syvitsky, 1991; Allen, 1997). A comparison for relative settling velocities of triaxial ellipsoids (denoted by L = long, I = intermediate, and S = short axis) is depicted in Fig. 2.4–6. (Matthews, 1991).

Shape effects are accentuated for disc-shaped particles such as expected for the soil clay fraction. The drag force acting on a settling disc with diameter X (and negligible thickness) is given by:

$$F_D = 8X\eta v \qquad \text{broadside}$$

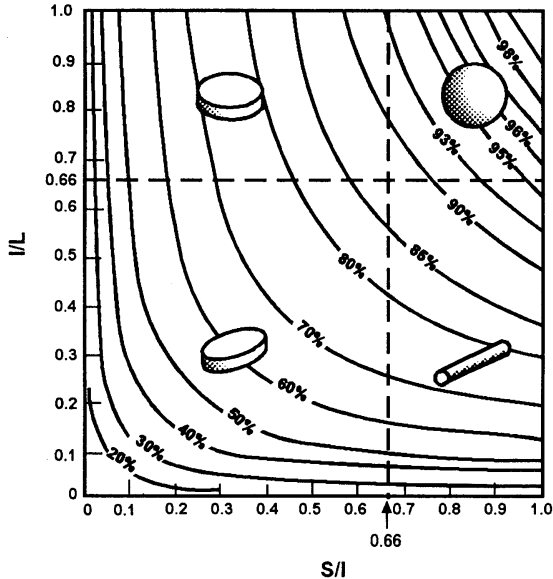$$F_D = (16/3)X\eta v \qquad \text{edgeside} \qquad [2.4-12]$$

Fig. 2.4–6. Settling velocity of triaxial ellipsoids as percentage of the velocity of equivalent sphere (Matthews, 1991).

for broadside and edgeside orientations. Averaging the drag force over all orientations of settling disc-shaped particles yields (Allen, 1997) $\bar{F}_D = 6X\eta v$. Considering the high degree of asymmetry of natural particles and their nonsteady settlement justifies the use of orientation-averaging, despite claims that particles settle with their maximum projected area perpendicular to the direction of motion (Komar & Reimes, 1978). The resulting settlement velocity of a disc is:

$$v_{disc} = [\alpha X^2 \pi g(\rho_s - \rho_l)]/24\eta \qquad [2.4\text{--}13]$$

where α is the ratio of disc thickness (h) to its diameter (X), defined as $\alpha = h/X$. The settling velocity of a disc relative to a spherical particle with a volume equal to the disc is $v_{disc}/v_{sphere} = 1.8\alpha^{1/3}$. The resulting velocity ratios are 0.83, 0.39, and 0.18 for $\alpha = 0.1, 0.01$, and 0.001, respectively. For a spherical particle with equal surface area as the disc, the ratio simplifies to $v_{disc}/v_{sphere} = 4.71\alpha$, and the resulting velocity ratios are 0.471, 0.0471, and 0.00471 for $\alpha = 0.1, 0.01$, and 0.001, respectively. Konert and Vandenberghe (1997) calculated and showed experimentally that a 2-μm-diameter sphere is equivalent to about a 7-μm-diameter clay plate with $\alpha = 0.036$. These calculations and experimental evidence highlight the need to consider particle shape in the interpretation of settlement data, especially for the soil colloidal fraction.

Electroviscous Effects. Viscous forces developing during settlement of clay particles distort the diffuse double layer and set up an electrical field opposing the motion. Allen (1997) shows that for surface-charge density, σ_e, and in the presence

of an electrical field, E, the Stokes settling velocity diminishes by the quantity $(X\sigma_e E)/3\eta$. The magnitude of this effect depends on electrolyte concentration in the suspension (e.g., the removal of soluble salts could enhance this effect). Siano (1979) found this effect to be negligible for polystyrene colloidal spherical particles.

Centrifugal Sedimentation. Centrifugal sedimentation is often used to separate submicron particles (where gravitational sedimentation is too slow). A particle in a centrifugal field is acted upon by a drag force and a centrifugal force. The force balance equation after attainment of equilibrium conditions is:

$$3\pi\eta X(dr/dt) = (\pi/6)(\rho_s - \rho_l)X^3\omega^2 r \qquad [2.4\text{--}14]$$

where r is the distance from the axis to the particle, dr/dt is outward velocity, and ω is the centrifuge angular velocity. Unlike gravitational sedimentation, the centrifugal settling velocity is not constant, but increases with increasing r. Rearranging and integrating Eq$_4$ [2.4–20] between initial settlement radius $r = r_1$ and final radius $r = r_2$ yields:

$$X = \sqrt{\frac{18\eta \ln(r_2/r_1)}{(\rho_s - \rho_l)\omega^2 t}} \qquad [2.4\text{--}15]$$

where t is the time for a particle of diameter X to settle from r_1 to r_2. Commercially available instruments combine centrifuge with either optical or x-ray attenuation methods (for sensing the particle density at various distances) or employ automatic pipette withdrawal with the centrifuge (Allen, 1997). Good results to <0.1 μm were confirmed (the claimed resolution is down to 0.01 μm).

Because the interest in this method invariably involves colloidal particles, it may be possible to extend the analysis to include disc-shaped clay particles by considering different forms of the drag force (left-hand side of Eq. [2.4–14]).

2.4.3.4 Pipette Method

2.4.3.4.a Principles

The pipette method is a direct sampling procedure. It depends on taking a small subsample by a pipette at a depth, h, at time, t, in which all particles coarser than X have been eliminated. Using Stokes' Law in the form of Eq. [2.4–4], settling times for the clay fraction (<2 μm) can be calculated for sampling at a given depth for a given temperature. Table 2.4–2 lists sampling times for the clay fraction for a 10-cm sampling depth at selected temperatures for the pipette technique. Tables 2.4–3 and 2.4–4 list sampling depths and times for various selected size fractions and specified settling times.

Experimental measurements with HMP solutions (Gee & Bauder, 1986) show the following relationships for solution viscosity and density:

$$\rho_l = \rho^\circ (1 + 0.630 C_s) \qquad [2.4\text{--}16]$$

Table 2.4–2. Settling times for 2-μm clay at various temperatures. Calculated for a 10-cm sampling depth in distilled water, 0.5 and 5 g L^{-1} HMP solutions, with a particle density equal to 2.60 Mg m^{-3}.

Temperature	Viscosity			Settling time		
	Distilled H$_2$O	0.5 g L^{-1} HMP	5.0 g L^{-1} HMP	Distilled H$_2$O	0.5 g L^{-1} HMP	5.0 g L^{-1} HMP
°C	——— 10^{-3} kg m^{-1} s^{-1}———			——— h ———		
18	1.0530	1.0553	1.0759	8.39	8.41	8.58
20	1.0020	1.0042	1.0238	7.99	8.00	8.16
22	0.9548	0.9569	0.9756	7.61	7.63	7.78
24	0.9111	0.9131	0.9310	7.26	7.28	7.42
26	0.8705	0.8724	0.8895	6.94	6.95	7.09
28	0.8327	0.8345	0.8508	6.64	6.65	6.78
30	0.7975	0.7992	0.8149	6.36	6.37	6.50

Table 2.4–3. Selected depths for 2-μm clay at specified times and temperatures, assuming a particle density of 2.60 Mg m^{-3} and dispersion of 0.5 g L^{-1} HMP solution.

Temperature	Viscosity	Sampling depth			
		4.5 h	5.0 h	5.5 h	6.0 h
°C	10^{-3} kg m^{-1} s^{-1}	——————— cm ———————			
20	1.0042	5.6	6.2	6.9	7.5
21	0.9800	5.8	6.4	7.0	7.7
22	0.9569	5.9	6.5	7.2	7.9
23	0.9345	6.0	6.7	7.4	8.1
24	0.9131	6.2	6.9	7.6	8.2
25	0.8923	6.3	7.0	7.7	8.4
26	0.8724	6.5	7.2	7.9	8.6
27	0.8532	6.6	7.4	8.1	8.8
28	0.8345	6.8	7.5	8.3	9.0
29	0.8166	6.9	7.7	8.4	9.2
30	0.7992	7.1	7.8	8.6	9.4

Table 2.4–4. Sampling times for 5- and 20-μm size fractions at a 10-cm sampling depth for pipette in 0.5 g L^{-1} HMP solution, for the temperature range 20 to 30°C for selected particle densities.

Temperature	5-μm particle size Particle density (Mg m^{-3})			20-μm particle size Particle density (Mg m^{-3})		
	2.4	2.6	2.8	2.4	2.6	2.8
°C	——————————— min ———————————					
20	87.7	76.8	68.3	5.5	4.8	4.3
21	85.7	75.0	66.7	5.4	4.7	4.2
22	83.7	73.2	65.1	5.2	4.6	4.1
23	81.7	71.5	63.6	5.1	4.5	4.0
24	79.9	69.9	62.1	5.0	4.4	3.9
25	78.0	68.3	60.7	4.9	4.3	3.8
26	76.3	66.8	59.3	4.8	4.2	3.7
28	73.0	63.9	56.8	4.6	4.0	3.5
29	71.4	62.5	55.6	4.5	3.9	3.5
30	69.9	61.2	54.4	4.4	3.8	3.4

where ρ_l is the solution density at temperature, t (g mL^{-1}); $\rho°$ is the water density at temperature, T, (g mL^{-1}); C_s is the concentration of HMP (g mL^{-1}) and

$$\eta = \eta° \ (1 + 4.25C_s) \qquad\qquad [2.4-17]$$

where η is the solution viscosity at temperature, t (10^{-3} kg m^{-1} s^{-1} [cpoise]), and $\eta°$ is the water viscosity at temperature (10^{-3} kg m^{-1} s^{-1} [cpoise]).

Equations [2.4–5] and [2.4–6] apply to HMP solutions in the range of 0 to 50 g L^{-1}. For tests with HMP solution concentrations in the range 0 to 5 g L^{-1}, <0.3% error in settling time results when the solution density is assumed to be that of pure water. A similarly low error results when 0.5 g L^{-1} HMP solutions are assumed to have the viscosity of pure water. However, settling-time errors as great as 2% result from not correcting for increased viscosity when using 5 g L^{-1} HMP solutions. Water densities and viscosities at various temperatures are available from Weast (1983).[1]

Particle densities should be known with a precision of at least ± 0.05 Mg m^{-3}. Settling-time errors in excess of 2% occur if particle densities are not known with at least this precision (see Table 2.4–3).

The pipette method is often used as a standard method from which other PSA methods are compared. This procedure has been adapted from Day (1965) and Green (1981).

2.4.3.4.b Apparatus and Reagents

1. Beakers—100 to 1000 mL; centrifuge bottles, both glass and plastic—250 mL
2. Centrifuges—low speed, about 1500 rpm, and high speed, about 12 000 rpm, with 250-mL bottles
3. Filter candle—porous ceramic tube, 0.05 MPa (0.5 bar) pressure rated
4. Shakers—horizontal reciprocating shaker, sieve shaker, wrist action shaker, holders for 250-mL centrifuge bottles on paint shaker
5. Cylinders—1000 mL (height of 1000-mL mark, 36 ± 2 cm)
6. Large (no. 13) rubber stoppers for 1000-mL cylinder
7. Stirrers—electric stirrers for mechanical mixing (available from Soil Test, Inc., Evanston, IL, or other source), hand stirrer made by joining a brass rod about 50 cm long to the center of a thin circular piece of perforated brass or plastic sheeting.[2] The circular plate should be cut to fit easily into the sedimentation cylinder. A 6-cm-diameter plate is normally adequate. If brass is used, place a wide rubber band around the edge of the brass sheeting to prevent scratching of the cylinder.
8. pH meter
9. Pipette rack-device to permit sliding the pipette laterally and lowering the pipette to a precise depth in the sedimentation cylinder (Clark, 1962; Day, 1965; see also Fig. 2.4–7)

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[1] Note that Weast (1983) reports viscosity in centipoise (cpoise). For conversion to SI units, 1 cpoise = 10^{-3} kg m^{-1} s^{-1}.

[2] Trade names are used in this chapter solely for the purpose of providing specific information. Mention of a trade name does not constitute a guarantee of the product, not does it imply an endorsement over other products not mentioned.

10. Lowy pipettes—25-mL capacity (available from Sargent-Welch Co., Skokie, IL, or other source)
11. Weighing bottles—beakers can be used
12. Set of sieves—square mesh with bronze wire cloth, 7.6-cm (3 in.) diameter with the following openings: 1000, 500, 250, 106, 53, or 47 μm
13. Reagents—hydrogen peroxide (~ 30%); 1 M NaOAc (adjusted to pH 5); citrate-bicarbonate buffer: prepare 0.3 M sodium citrate (88.4 g L^{-1}) and add 125 mL of 1 M sodium bicarbonate (84 g L^{-1}) to each liter of citrate solution; sodium dithionite (hydrosulphite); saturated NaCl solution; 10% NaCl solution; 1 M AgNO$_3$; 1 M BaCl$_2$; acetone; Na-HMP, 50 g L^{-1} stock solution; 1 M CaCl$_2$; 1 M HCl
14. Electric oven

2.4.3.4.c Method

Pretreatment. Several pretreatments are described as follows.

Removal of Carbonates and Soluble Salts. Weigh a small portion of the <2-mm fraction of air-dry soil into a 250-mL centrifuge bottle (10 g for clays, 20 g for loams, 40 g for sandy loams and loamy sands, and 80 g for sands). Weights are optional, but these are generally suitable if clay samples are required for mineralogy. Add approximately 100 mL of water, mix, and add 10 mL of 1 M NaOAc (adjusted to pH 5). Centrifuge (about 10 min at 1500 rpm) until the supernatant is clear, and then pour it off. Wash the soil twice by shaking with 50 mL of water, centrifuging, and discarding the supernatant if it is clear. If the centrifugate is not clear (as is often the case for soils containing high amounts of soluble salts and soils containing gypsum), further washing may be necessary. Washing through a filter candle to remove salts is a permissible substitute for centrifugation, but this procedure takes considerably longer than centrifugation. Check for salts by testing with AgNO$_3$ for Cl$^-$ and BaCl$_2$ for SO$_4^{2-}$.
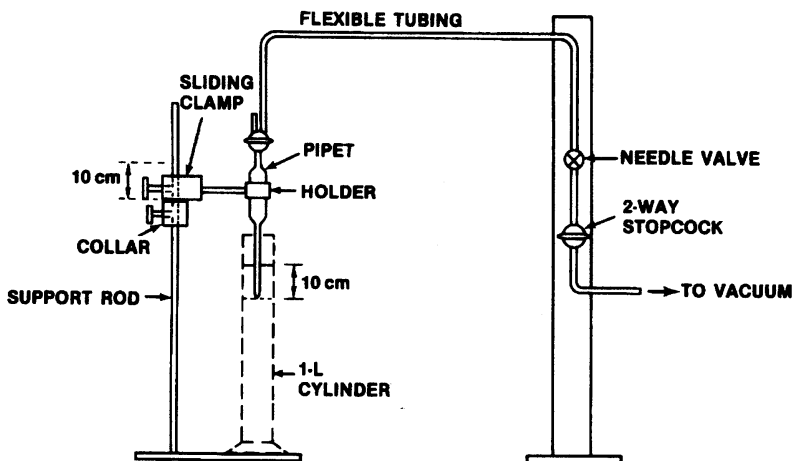

Fig. 2.4–7. Schematic diagram of pipette stand and apparatus for sedimentation analysis.

Removal of Organic Matter. After carbonate removal, add 25 mL of water to the soil in the centrifuge bottle, and shake on a wrist-action shaker. Transfer samples containing high amounts of organic matter (>5%) to 1000-mL beakers. Add 5 mL of (H_2O_2) to the soil suspension, stir, cover, and observe closely for several minutes. If excessive frothing occurs, cool the container in cold water. Add more H_2O_2 when the reaction subsides. Note that MnO_2 decomposes H_2O_2 and SO if present in measurable amounts; steps should be taken to complex or remove before peroxide treatment. Heat to 90°C when frothing has ceased, remove cover, and evaporate excess water (do not take to dryness). Continue peroxide and heat treatment until most of the organic matter has been destroyed (as judged by the rate of reaction and the bleached color of the sample). Rinse down the sides of the reaction vessel occasionally. Heat for about an hour after the final addition of peroxide to destroy excess peroxide. Transfer the sample to a 250-mL glass centrifuge bottle.

Removal of Iron Oxides. Add citrate-bicarbonate buffer to the peroxide-treated sample in the centrifuge bottle to bring the total volume of solution to approximately 150 mL. Shake to disperse the soil. Add 3 g of sodium dithionite ($Na_2S_2O_4$) gradually, as the sample may froth. Put the bottle into a water bath at 80°C and stir the suspension intermittently for 20 min. Remove the sample from the bath, add 10 mL of saturated NaCl, mix, centrifuge, and decant off the centrifugate. It may be combined with subsequent centrifugates, if any, and analyzed for dithionite-extractable Fe, Al, or Mn. If the sample is completely gray (gleyed), proceed to the next step. If a brownish color remains, repeat the previous step. Wash the sample once with 50 mL of citrate-bicarbonate buffer plus 20 mL of saturated NaCl (shake, centrifuge, and decant). Wash the sample twice with 50 mL of 10% NaCl, then twice with 50 mL of distilled water. If the wash solution is not clear, transfer the sample to a plastic centrifuge bottle and centrifuge at high speed. If this fails to yield clear centrifugate, add acetone, warm the sample, and recentrifuge. Add 150 mL of water, shake the sample, and check the pH. It should be above pH 8 if the soil is Na-saturated. Transfer the suspension to a 1-L shaker bottle, add 400 mL of distilled water and 10 mL of HMP (dispersant) stock solution, and shake overnight on a horizontal shaker.

Separation of Sand Fractions. Pour the suspension through a 53-μm (270-mesh) sieve into a 1-L sedimentation cylinder. A 20-cm-diameter (8-in.) sieve is placed in a large funnel held by a stand above the cylinder. Tap the funnel gently and wash the sand thoroughly on the sieve. A soap solution placed on the sieve will aid in wetting the fine screen. Collect the washings in the cylinder. Transfer the sand to a tared beaker or aluminum weighing dish, dry (105°C), and weigh.

Transfer the dried sand to the nest of sieves arranged from top to bottom with decreasing size in the following order: 1000-, 500-, 250-, 106-, 53-μm, and pan. Shake the sieves on a sieve shaker. A 3-min shaking time is usually adequate. Weigh each sand fraction and the residual silt and clay that passed through the 53-μm (270-mesh) sieve. A weighing precision of 0.01 g is adequate.

Determination of Silt Fractions. The 20- and 5-μm fractions can be determined by pipette by following the procedure outlined in the next section for clay and using Eq. [2.4–8] or Table 2.4–3 for determining the required settling times.

Determination of Clay (<2 μm). Place the cylinder containing the silt and clay suspension in a water bath; add 10 mL of HMP solution and make up to 1 L volume with distilled water; cover with a watch glass. Let the suspension stand at least several hours to equilibrate.

After thermal equilibration, stir the suspension thoroughly with a hand stirrer for at least 30 s using an up-and-down motion. Note the time at completion of stirring and the temperature of the water bath. It is convenient to complete the stirring of adjacent suspensions at intervals of about 3 min. An alternative to hand stirring is stoppering the sedimentation cylinder and shaking end-over-end for 1 min.

After the appropriate time interval after cessation of stirring (see Tables 2.4–1 through 2.4–3), lower the closed Lowy pipette carefully to the appropriate depth, turn on the vacuum, and withdraw a 25-mL sample in about 12 s (see Fig. 2.4–7). A device for controlling the vacuum is required.

Discharge the sample into a tared and numbered weighing bottle, beaker, or aluminum dish. Rinse the pipette with distilled water and add the rinse water to the clay suspension in the weighing bottle. Evaporate the water, dry the clay at 105°C, cool in a desiccator, and weigh.

Determining the Weight of Treated Soil. Add 10 mL of 1 M CaCl$_2$ and 1 mL of 1 M HCl to the suspension remaining in the cylinder to prevent CaCO$_3$ formation. Siphon off the clear solution after flocculation has occurred. Transfer the soil from the cylinder to a tared beaker, evaporate, dry at 105°C, and weigh.

Differences between original soil weight and weight found in the cylinder are attributed to pretreatment soil loss, solution loss, sieving loss, and sample removal for pipette-sieving analysis. The total oven-dry weight of the treated sample is used as the basis for calculating the size fraction. The total oven-dry weight can be expressed as:

$$W_s + W_p + W_r = W_t \qquad [2.4\text{--}25]$$

where W_s is the oven-dry weight of the sand fraction, W_p is the corrected oven-dry weights of pipette samples, W_r is the corrected oven-dry weight of residual silt and clay, and W_t is the total weight of treated sample. W_p and W_r are corrected by subtracting the weight of the dispersing agent (Table 2.4–4).

Calculations. Table 2.4–5 shows how the pipette method is used to determine size-fraction percentages using a 25-mL pipette.

Table 2.4–5. Example calculations of three particle-size percentages using a 25-mL pipette.

Particle size	Sample weight	Concentration	Corrected concentration†	Percentage less than‡
mm	g	g L^{-1}		%
0.020	0.114	4.56	4.06	39.8
0.005	0.073	2.92	2.42	23.7
0.002	0.057	2.28	1.78	17.4

† Dispersing agent concentration = 0.5 g L^{-1}.
‡ Based on oven-dry weight of treated sample, W_t = 10.21 g.

2.4.3.4.d Comments

A modified pipette procedure using a shorter sedimentation flask, called a *fleaker* (combination flask and beaker), has been used by Indorante et al. (1990). Fleakers cost less than sedimentation cylinders and allow for quicker sample preparation. According to Indorante et al. (1990), fleakers take up less space than 1-L sedimentation cylinders, thus accommodating large batch processing as might be needed in government, university, or commercial soil testing laboratories.

Flocculation of clay from suspension has been observed in soils containing large amounts of gypsum (Kaddah, 1975; Hesse, 1976; Rivers et al., 1982). Flocculation is recognized by a distinct separation of clear liquid and suspended clay (flocculated clay often has the appearance of a cloudy gel-like precipitate). Removal of soluble salts (Section 2.4.2.5) helps prevent flocculation in most soils. However, gypsum, having a low but measurable solubility, can cause flocculation by replacing Na with Ca. Procedures for removing gypsum are available (Rivers et al., 1982). Options for removing gypsum include adding Ba (Hesse, 1976) or increasing the concentration of HMP dispersant (Kaddah, 1975). Flocculation must be prevented for sedimentation analysis (pipette, hydrometer) to provide meaningful results.

Errors in PSA values using the pipette analysis are mainly associated with sampling and weighing. With care, clay fractions can be determined with a precision of ± 1% (w/w) using pipette procedures. Because it is a direct weighing method, the pipette method is probably superior to other sedimentation methods, particularly at low clay contents, where other methods, such as the hydrometer method, have lower sensitivities.

2.4.3.5 Hydrometer Method

Particle-size analysis can be done conveniently with a hydrometer, which allows for nondestructive sampling of suspensions undergoing settling. The hydrometer method provides for multiple measurements on the same suspension so that detailed particle-size distributions can be obtained with minimum effort. The hydrometer method outlined is modified from Day (1965) and ASTM (2000d).

2.4.3.5.a Principles

The hydrometer method, like the pipette method, depends fundamentally upon Stokes' Law, which for the hydrometer may be written as

$$X = \theta t^{-1/2} \qquad\qquad [2.4\text{--}18]$$

where θ is the sedimentation parameter and $t^{-1/2}$ is a function of the hydrometer settling depth, solution viscosity, and particle and solution density. This relationship follows from Eq. [2.4–4] by rearranging terms such that

$$X = \left[\frac{18\eta h'}{g(\rho_s - \rho_l)} \right]^{1/2} t^{-1/2} \qquad\qquad [2.4\text{--}19]$$